

Recent Approaches in Guar Gum Hydrogel Synthesis for Water Purification

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Abstract

Guar gum based hydrogels have evoked great attention in research as well as in industry for exploring miscellaneous applications in the field of water purification, medicine, agriculture, explosives, cosmetics, textile, paper and food to name a few. In this article, latest modifications for developing guar gum based hydrogels and composite materials for water purification application are extensively reviewed. Regenerative nature of Guar gum hydrogels makes it a better choice for treating waste water as well as other value added applications. Moreover, non-ionic nature makes guar gum an acceptable material for further modifications. In this article, we also presents brief discussion on structure and properties of guar gum.

Keywords: Guar gum; hydrogel; composites, dye adsorption; metal ion adsorption; water purification.

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Introduction

Over past 50 years hydrogel technology has come in to view as a new prevalent technology which may be going to discover more applications in different fields of research[1]. Hydrogels have evolved with inherent advantages beyond continuing technologies like diffusion and adsorption[2]. Due to industrial evolution and population expansion, need for more drinkable water as well as other sustainable materials from natural resources including gums and natural biomass has become an important issue[3–8]. The inattentive behavior of humans towards environment has majorly affected the condition of water by contaminating it with inadequate quantities of pesticides, drugs, dyes and other pollutants[9–13]. Hydrogels may act as new emerging technology that is being researched for water remediation[14]. Self-assembly of molecules is considered as the unique property, which is essential for the emergence or advancement of any material and hydrogel also show self-assembling nature in water[15]. Hydrogels exhibit most fragile three dimensional structure, soft nature, high water soaking capacity and porosity which makes it extremely suitable for loading water soluble bodies[16]. Furthermore, hydrogels are the biodegradable gels with interconnecting pores which play essential role in adsorption process and for confining more aquatic pollutants[3, 9], its porosity can be permanently achieved by following modifications: (a) by cryogelation[17], (b) integrating nanoparticles[4, 18], (c) by lyophilization (drying-freeze of swollen hydrogel)[17], (d) by grafting mechanism[19–21] and (e) by cross-linking polymerization[22]. Natural hydrogels can be replaced by artificial hydrogels having long life, high adsorption capacity, more mechanical strength and high swelling nature[23]. Herein, this review article, we have focused on guar gum derived hydrogel composites for waste water treatment. The structure and properties of guar gum and composites have also been discussed in this article.

Guar gum

A drought resistant plant named *Cyamopsis tetragonoloba* belongs to family leguminosae is an important agrochemical, derived from the endospermic part of guar seed mostly cultivated in Pakistan and India from ancient time is now becoming useful for many purposes[24]. It is a source of guar gum or guaran. Before 1940, guar gum has been reviewed in a limited extend[25]. It has achieved popularity in the field of science roughly around 1940s and 1950s and was used as industrial product in 1943[26]. Guar gum has strong hydrogen bonding tendency in water, that's why it acts as best thickener and stabilizing agent in various chemical processes. The use of this gum has increased tremendously in last few years specially in cosmetics, oils, paints, as adjunct in paper making, emulsifier, suspending and wound-healing agent[25]. The yellowish-white, natural, non-ionic and unprocessed guaran is a reserved part of the cluster bean which is essential for the germination of seed. On coming in contact with water like alginic acid, its inter-molecular chain creates a strong bond with surrounding hydroxyl groups which is necessary for attaining maximum viscosity potential in cold water[27]. Cluster bean is not considerably influenced by the pH, but at 3.5 pH it exhibits low viscosity potential and in between 6-9 it shows maximum viscosity potential that means over a broad pH range it resist with unchanging viscosity rate[28]. The development of gum derivatives is often necessary for fulfilling specific application requirements, derivatization of the unprocessed or virgin gum escort the desired and modified properties of the gum without ignoring inherent values up to the mark[29]. No doubt, chemical modifications like grafting, cross-linking, etherification and carboxylation makes it suitable for elevating potential applications in various fields of science but sidewise care should be taken to remove its inborn deficiencies so that we can use it for long term applications[30][31]. A raw structure with high molecular weight can be tailored to obtain desired range of properties for making it suitable for the certain applications.

A number of derivatizations have been made on guar gum to elaborate its use in various fields like food industry[32], explosives[33], flocculating agent[34][35][36], agriculture[37], pollutant removing agent[38][39][40][41], paper industry[42], cosmetics[43], drug delivery[44] and in pharmaceuticals[45].

Structural compositions of guar gum

Guar gum is a natural non-ionic polysaccharide having high molecular weight drawn out from the endosperm portion of seed which basically consist of straight chain of D-mannose subunits united by β (1-4) glycosidic bond and D-galactose subunits linked together by (1-6) glycosidic bond at each alternate positions[46]. The endosperm layer of guar kernel predominates the presence of galactose and mannose sub-unites which are collectively called as galactomannan units[47]. The ratio of galactose to mannose units has been reported as 1:2 and in range of 1:1.6 to 1:1.8[48]. **Figure 1** shows the enlarged structure of endosperm wherein subunits are linked together at different positions to form a large polymeric chain. The core network of guar gum resembles like other polysaccharide gums but its elemental composition includes complex galactomannan units (75-85%), 8-14% of moisture content, 5-6% of protein, 2-3% of fibers and 0.5-1.0% of ash[46].

Physical and chemical properties of guar gum

The properties like swelling, solubility and hydrogen bonding making tendency are maximum in polar solvents rather than non-polar. Herein this review, we have tried to study all physical and chemical nature of the gum like dissolution, rheology, effect of pH and temperature, viscosity and most important one hydration rate which can be reduced by adding certain salts and binding sugars like sucrose [49].

Viscosity

It is a state of being thick, which depends upon percentage hydration of guar gum in cold water[50]. Guar gum is commonly thixotropic in nature because, on continuous stirring its gels come in liquid form. Moreover, high thixotropic behavior can be seen in case of more than 1% of guar gum in aqueous solution[51]. Its viscosity equally depends upon temperature, pressure, pH and amount of continuous agitation during synthesis[28].

Rheology study

Rheology gives the idea of deformation and flow of the viscous fluids when any external stress is applied. Guar gum exhibits pseudo plastic behavior which in rheology called as non-Newtonian behavior of gels[52]. According to shear-strain theory, viscosity of fluids undergo reduction with increase in shear-strain and this theory confirms the shear-thinning behavior of the different polymeric gels[53].

Strong hydration rate

Hydration is a physical phenomenon in which gum creates the cementing bonding with the water molecules[54]. Hydration rate dynamically depends upon time and size of the gum particles[33]. Rate of hydration increases with small particle size that means very fine powder of guar gum is required for getting quick viscosity at initial stage[55].

Strong hydrogen bonding

Strong hydrogen bonding in guar gum is because of the presence of hydroxyl groups[29]. For hydrogen bonding, a strong electronegative atom with heteronuclear dipole moment is required to complete dipole-dipole and attractive electrostatic interactions[56]. Guar gum shows strong

bonding with hydrated molecules and cellulose derivatives. Importantly, the stability of bonds decreases by steric hindrance caused due to substitution of –OH groups of gum by hydroxypropyl groups[57].

Effect of temperature

Temperature equally affects the hydration rate and viscosity of the fluids. Guar gels attain higher viscosity at high temperature but long term heating may also cause degradative effect. They also show high rate of hydration in warm water whereas cold water permits the gum to hydrate slowly[58]. The reported temperature for attaining maximum viscosity is about 25-40° C wherever for 0.5% guar solution, 25° C temperature gives higher viscosity than the temperature of 37° C and at constant temperature, 0.5% gum solution behaves as Newtonian system[59].

Effect of pH

Because of uncharged and non-ionic behavior, guar gum exhibits stable properties for wide range of pH. Different pH solutions do not affect the final viscosity but it may affect the hydration rate, and fastest hydration rate can be found at pH 8 which may drop at more than pH 10 or less than 4[60]. Considerably, maximum adsorption for various pollutant is shown by the guar gum based hydrogels at pH 10[61].

Effect of concentration

High viscosity is essential for fulfilling food applications and guar gum forms deep thick solution at low concentration[62]. Viscosity is just proportional to the concentration and size of the gum particles and this is because of water and galactose chain interaction. Increase in these side chain interactions entangles the more viscous behavior of gum solution[63].

Guar gum based hydrogels for water purification

One of the possible mechanisms followed in water purification is adsorption. For this, numbers of fillers and chemical modifications have been investigated to achieve required application in water remediation program[64],[65], [66], [67], [68], [69], [70],[71],[72]. **Table 1** shows different types of modified guar gum based hydrogels adsorbents material for water pollutants removal. Hydrogels based on guar gum-polyacrylamide (GG-Poly-AAm) and modified guar gum-polyacrylamide m-(GG-Poly-AAm) were used for evaluating removal percentage of azure B and crystal violet dyes[65]. Methylene bis-acrylamide and glutaraldehyde were used as cross-linkers for getting well linked network. In this work, saponification of prepared hydrogel was performed in strong basic media to improve compatibility for dye sorption. Moreover, alkaline hydrolysis of m-(GG-Poly-AAm) led to the generation of carboxylic groups which enhanced the anionic moiety of the hydrogel network.

The differential scanning calorimetry (DSC) pattern of GG-Poly-AAm (neutralization equivalent (NE) =0), m-GG-Poly-AAm1 (NE= 426.2), m-GG-Poly-AAm2 (NE=294.4) and m-GG-Poly-AAm3 (NE=222.0) were investigated. The melting transition peak for GG-Poly-AAm was spotted at 273 °C, which was slowly diminished in m-GG-PolyAAm because of formation of carboxylic groups from amide functionality and totally disappeared in m-GG-Poly-AAm3 which again assured the formation of more –COOH groups inside the gel. TGA analyses showed the mass loss of the samples which was about 5 % at starting and then increased to 30-35 % at 230-350 °C assigned to the loss of –OH groups of guar gum. The hygroscopic nature of m-GG-Poly-AAm samples was confirmed by higher mass loss percentage at initial stage which was then equivalent to the GG-Poly-AAm gel at intermediate stage. Kinetic study displayed that system followed the

second order kinetics and thermodynamic evaluation supported the exothermic adsorption of dyes on mGG-Poly-AAm2 surface.

Methylene blue dye was removed by Sharma et al. using polyaniline based guar gum/acrylic acid hydrogel[66]. They synthesized guar gum cross-linked polyacrylic acid (GG-cl-Poly(AA)) hydrogel by solvent evaporation which was further used for the preparation of guar gum cross-linked polyacrylic acid with interpenetrating network of polyaniline (GG-cl-Poly(AA-ipn-aniline)). Similar method was followed in acidic conditions where GG-cl-Poly (AA-ipn-aniline) hydrogel was treated with 0.5N solution of HCl to form doped-GG-cl-Poly (AA-ipn-aniline) hydrogel. Molecules of methylene dye were separated from different gel mixtures, their dye uptake efficiency was investigated in detail. It was mentioned that percentage removal for dye may vary with different parameters. With passage of time, methylene blue percentage removal was increased to 52.98%, 54.68 % and 61.83% for doped-GG-cl-Poly (AA-ipn-aniline), undoped-GG-cl-Poly (AA-ipn-aniline) and GG-cl-Poly (AA) hydrogels respectively (**Figure 2a**). **Figure 2b** shows that dose of 300 mg was optimized for maximum removal percentages of 64.4 %, 56.4 % and 54.9 % for GG-cl-Poly (AA), undoped-GG-cl-Poly (AA-ipn-aniline) and doped-GG-cl-Poly (AA-ipn-aniline) hydrogels respectively. The maximum methylene blue adsorption was found at pH 10, dye adsorption was higher at high pH, poor adsorption at low pH because of high repulsive electrostatic interactions between dye and hydrogel samples (**Figure 2c**). The high temperature conditions were resulted in increased porosity and swelling, hence adsorption was enhanced with temperature (**Figure 2d**). At 70°C, adsorption efficiency was 69.15% for GG-cl-Poly (AA) hydrogel whereas undoped and doped hydrogel samples exhibited the efficiency of 64.22 % and 62.60 % respectively. The rough surface of cross linked hydrogels (**Figure 3b-d**) was observed

in comparison to smooth surface of pure guar gum (**Figure 3a**). The roughness of doped hydrogel surface was more intense than undoped hydrogel surface.

Thombare et al. developed hydrogel of guar gum cross linked with borax (GG-cl-B) at different concentrations[67]. The developed hydrogel was applied for the adsorption of aniline blue (AB) dye from water. The synthesis of GG-cl-B is clearly represented in **Figure 4**, where boron was believed to make bond with different oxygen atoms of hydroxyl groups[73].

Distinct flake like morphology (**Figure 5a**) and a compact smooth surface (**Figure 5b**) was reported for pure gum powder and guar gum film respectively[74]. But for GG-cl-B hydrogel, an extraordinary change was observed, the surface morphology changed to porous scaly structure which attributes more fluid diffusion (**Figure 5c**). Guar gum represented three different weight loss zones of 25–270 °C, 270–330 °C and 330–562°C whereas GG-cl-B hydrogel showed four different weight loss zones of 25–280 °C, 280–330 °C, 330–508 °C and 508–600 °C. Both samples showed maximum weight loss in second zone which were nearly about 54 % and 34 % for guar gum and GG-cl-B hydrogel respectively.

Variety of modifications has been done for the removal of heavy metal ions. Among all, grafting and integration of nanoparticles are considered as best techniques for developing best adsorbent material[68]. A low cost guar gum was used as a backbone for making novel hydrogel adsorbent material for sorption of Cr (VI) metal ion from water. The incorporation of bentonite nanoclay increased the mechanical strength and hydrophilicity of the prepared composite hydrogel. This hydrogel composite had achieved the removal percentage of 97.8% at pH 2.0.

Figure 6a shows the stepwise synthesis of guar gum/bentonite hydrogel composite (CPGB) started with free radical generation from initiator (potassium peroxydisulfate), followed with generation of monomers and gum radicals with the addition of N, N-methylenebis acrylamide as cross linker.

Finally, polymerization with bentonite nanoparticles was carried out to produce guar gum/bentonite hydrogel composite. **Figure 6b** represents the repeated evaluation of swellability and de-swellability of hydrogels at pH of 2.0 and 8.0. This study confirmed the good reversibility of hydrogels after 3 to 4 cycles of high and low pH and best mechanical strength was shown by the bentonite incorporated guar gum composite hydrogel (CPGB4) (guar gum = 1wt%, bentonite = 2wt% and monomer ratio (acrylic acid: hydroxyethyl methacrylate) = 10:1) than copolymer hydrogel (CP4) (guar gum = 0wt%, bentonite = 0wt% and monomer ratio (acrylic acid: hydroxyethyl methacrylate) = 10:1) and guar gum incorporated copolymer gel (CPG4) (guar gum = 5wt%, bentonite = 0wt% and monomer ratio (acrylic acid: hydroxyethyl methacrylate) = 10:1). Importantly, there was a loss in crystal behavior of guar gum and clay particles because of the attractive and repulsive interactions residing in between the functional groups of the gum and bentonite clay particles presented inside the polymeric gel (water + guar gum).

In another work, grafting of acrylamide was carried out to synthesize guar gum based hydrogel for the removal of hexavalent chromium ion [70]. The polymerization technique was used for the grafting of acrylamide onto the guar gum using redox system of potassium bromate and thiourea dioxide. In final step, prepared guar gum based polyacrylamide grafted polymer was cross-linked with glutaraldehyde to produce the hydrogel. The prepared guar gum grafted polyacrylamide hydrogel was utilized for the removal of chromium hexavalent ion with maximum adsorption capacity of 588.24 mg g⁻¹.

The synthesis of guar gum based polyacrylamide grafted copolymer is shown in the **Figure 7a**. The prepared guar gum based polyacrylamide grafted copolymer was further cross linked with glutaraldehyde for the formation of cross-linked hydrogel as represented in the **Figure 7b**. Furthermore, sorption study revealed that amount of adsorbed Cr (VI) ion depends upon the extent

of grafting in prepared hydrogel (**Figure 7c**). A novel guar gum based hydrogel was synthesized by Chauhan et al. for the sorption of Cu (II) ions using clean and effective acid hydrolysis[71]. Depolymerized guar gum and domestic guar gum were oxidized with the help of nitrogen oxide NO_x (oxidant) and then samples were cross-linked with N, N-methylene bisacrylamide. Maximum adsorption capacity of 125.893 mg g⁻¹ was reported after 2 hours at 40 °C using 20 ppm of Cu (II) solution.

Conclusions

In this article, we have tried to explore structure, chemical and physical aspects of guar gum with prime focus on its application as pollutant removing agent in water purification. It is a natural, harmless, renewable, low cost, highly viscous and pH compatible polysaccharide which may easily blend into supportable products. All these properties of guar gum provide an edge to other similar derivatives of this field. Guar gum is now being considered as one of the most promising potential candidate to explore better outcomes in possible adsorption techniques and can act as preferred choice for different areas of research interests.

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Figures:

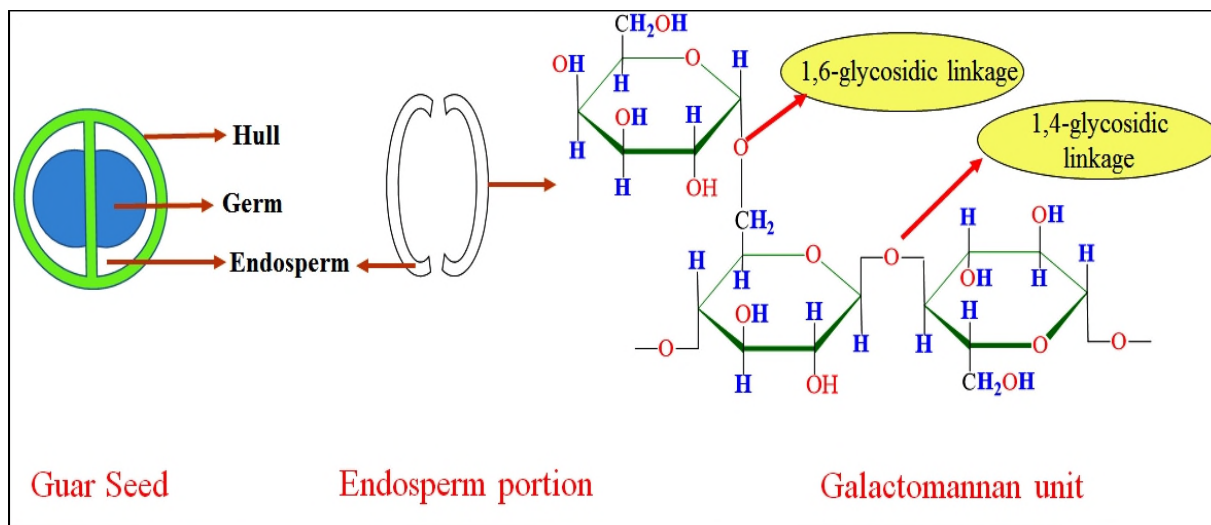


Figure 1. Showing endospermic part of guar seed which is made up of galactomannan units

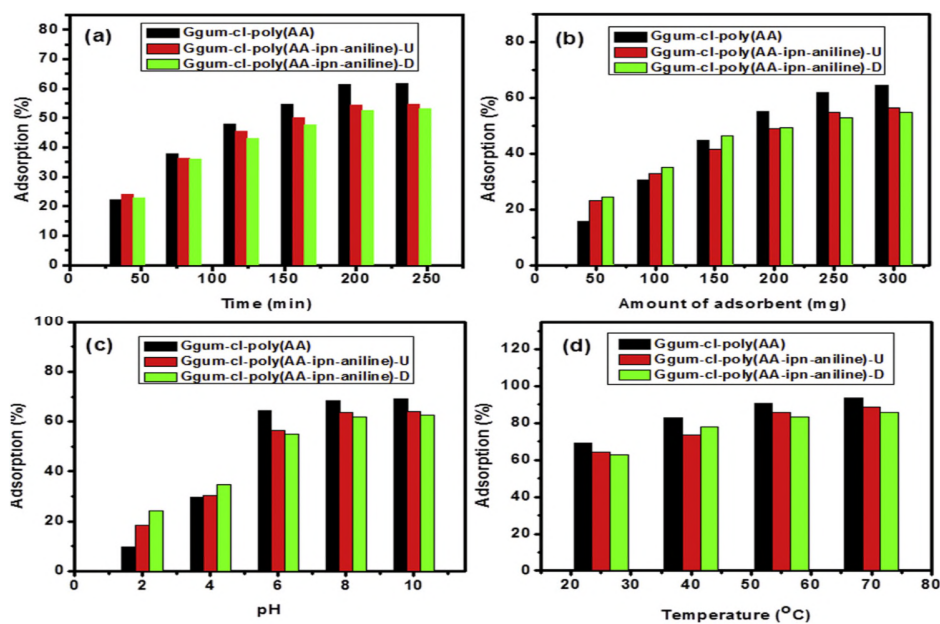


Figure 2. Different adsorption parameters explaining (a) effect of time, (b) effect of dose, (c) effect of pH and (d) effect of temperature [66]. Reprinted with permission from [66]. Copyright 2015 Elsevier.

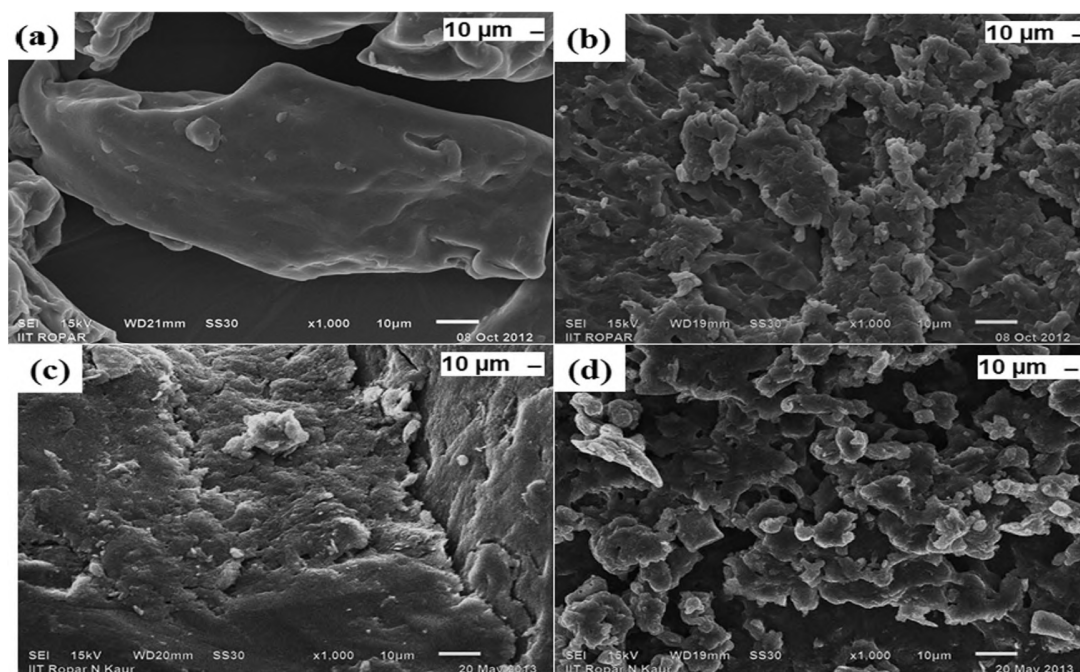


Figure 3. SEM images of (a) guar gum (b) GG-cl-Poly(AA) (c) undoped-GG-cl-Poly(AA-ipn-aniline) (d) doped-GG-cl-Poly(AA-ipn-aniline) [66]. Reprinted with permission from [66]. Copyright 2015 Elsevier.

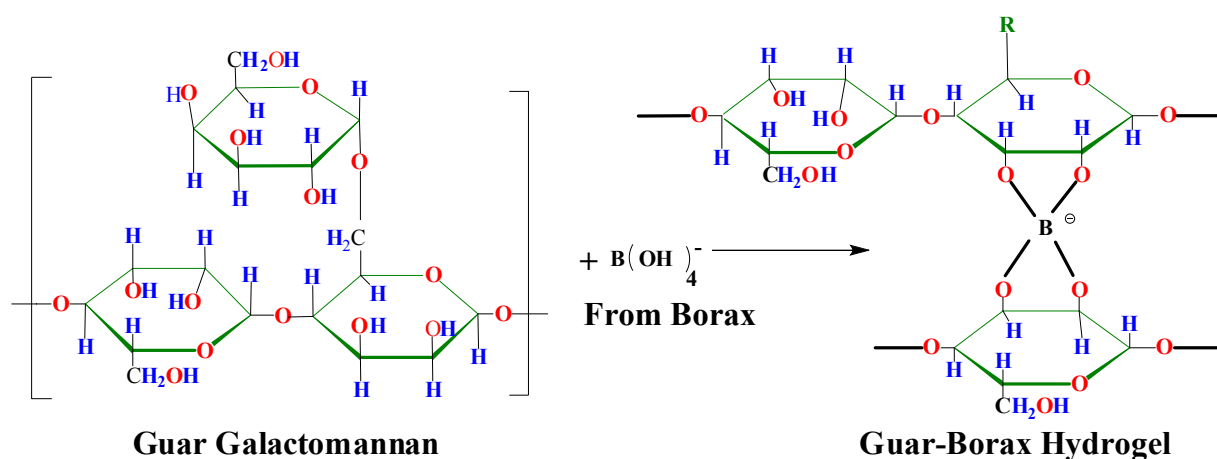


Figure 4. Schematic representation of synthesis of borax cross linked guar gum hydrogel (GG-cl-B)[67]. Reproduced with permission from [67]. Copyright 2017 Elsevier.

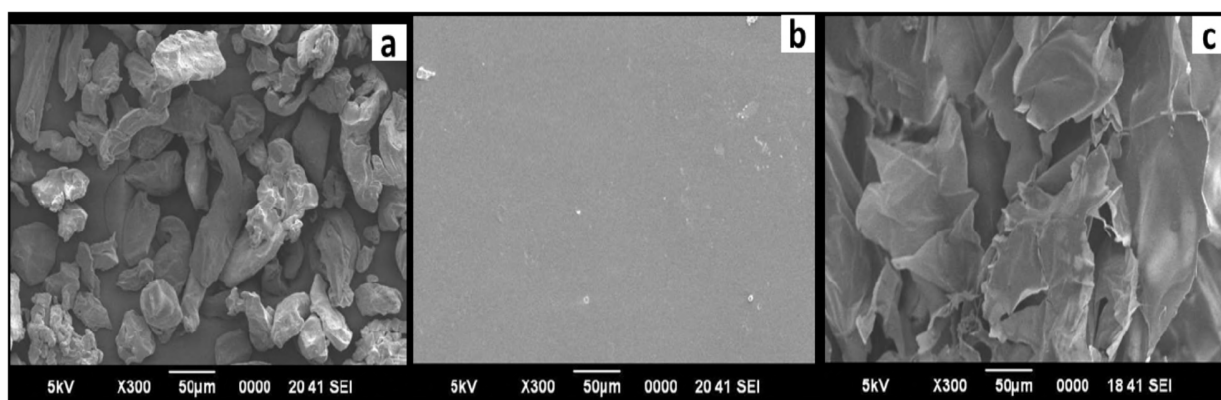


Figure 5. SEM images of **(a)** guar gum powder, **(b)** guar gum film, **(c)** GG-cl-B hydrogel[67].

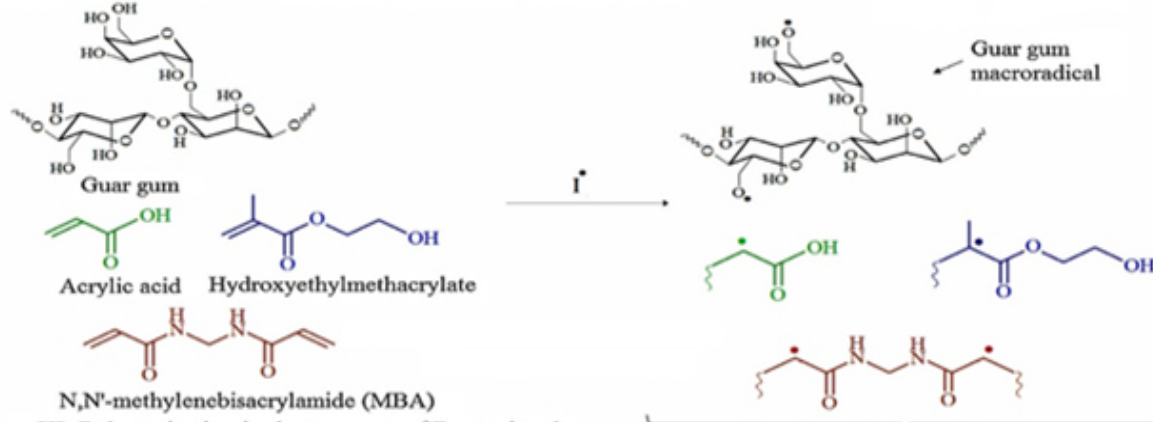
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Step-I: Generation of free radical from initiator



(a)

Step-II: Generation of guar gum macroradical and monomer radicals



Step-III: Polymerisation in the presence of Bentonite clay

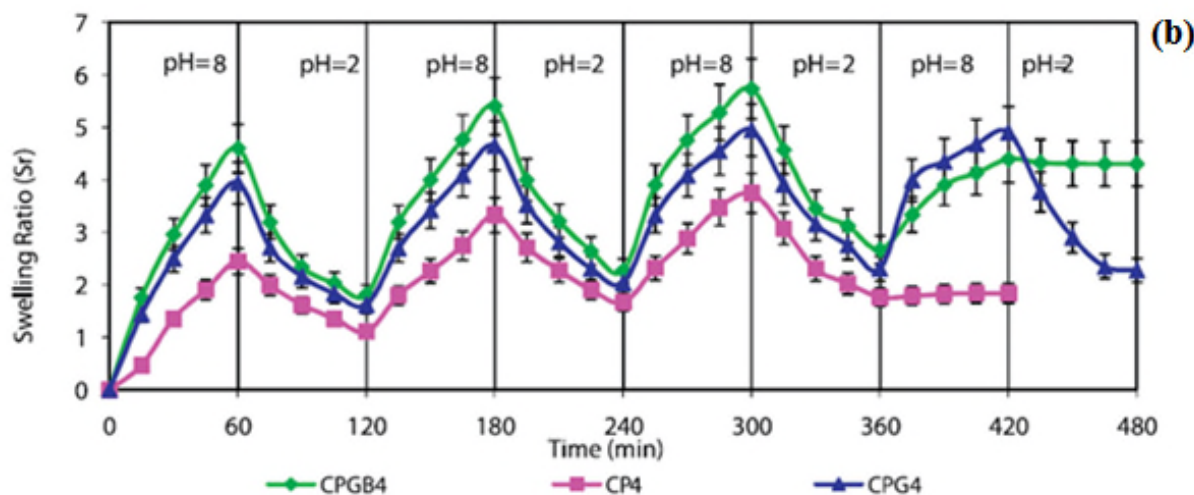
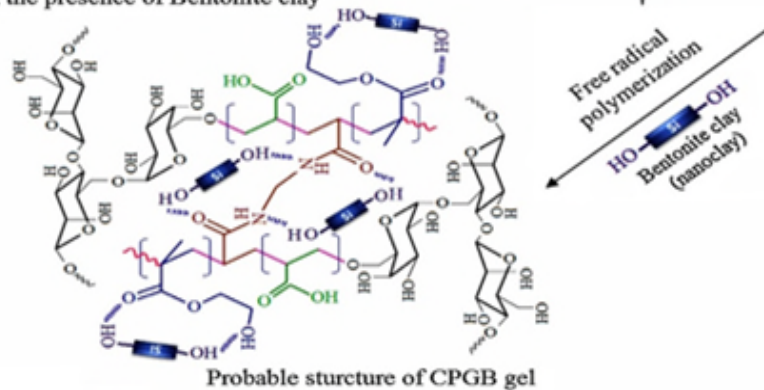


Figure 6. (a) Possible scheme for the synthesis of guar gum/bentonite composite hydrogel, (b) reversibility study of hydrogels with respect to time [68]. Reprinted with permission from [68]. Copyright 2016 Elsevier.

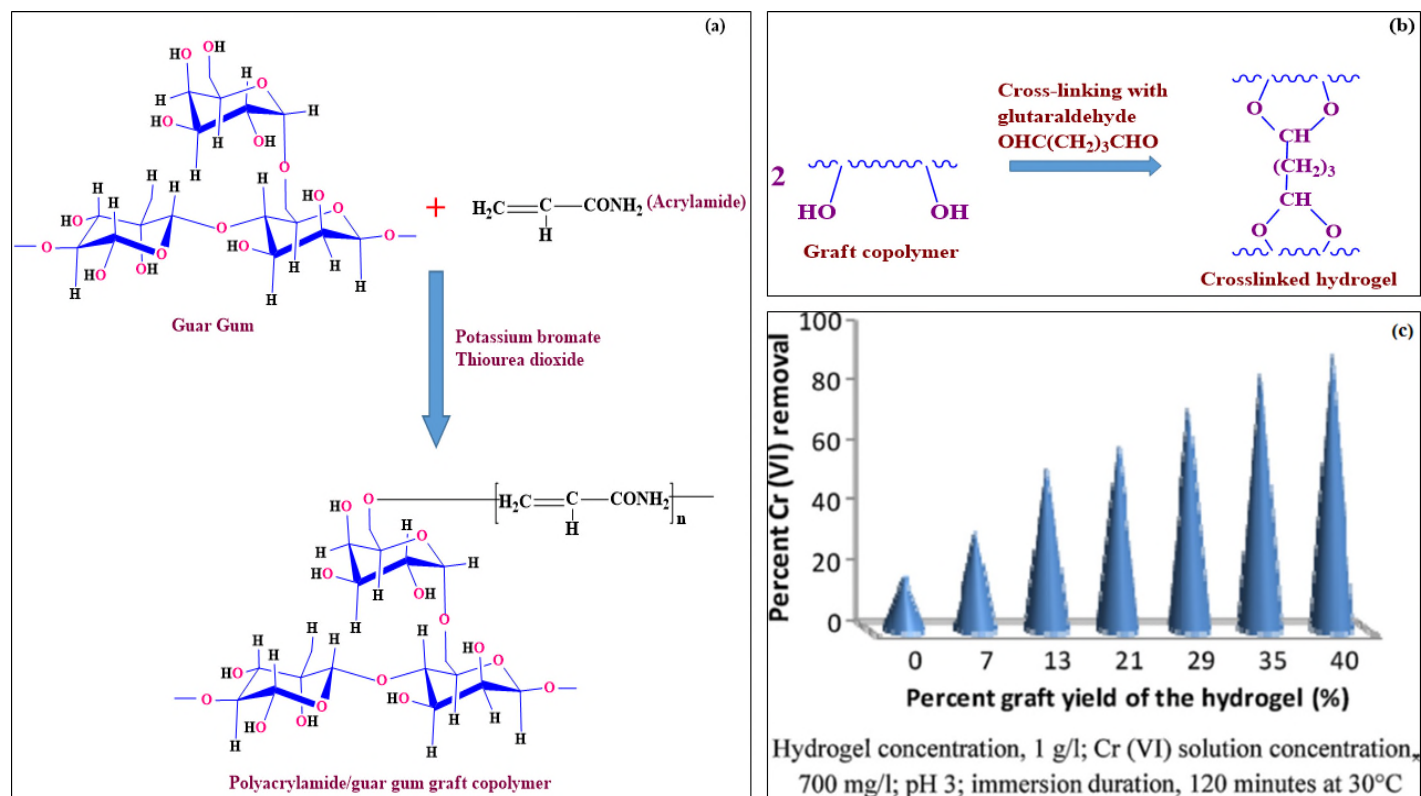


Figure 7. Possible mechanism for (a) synthesis of guar gum based polyacrylamide grafted copolymer, (b) crosslinking of guar gum based polyacrylamide grafted copolymer with glutaraldehyde and (c) effect of grafting on Cr (VI) ion adsorption [70]. Reprinted with permission from [70]. Copyright 2011 Elsevier.

Table 1. Guar gum based hydrogels for removal of organic and inorganic pollutants.

| Sr. No. | Hydrogel Composites | pH | Organic & Inorganic Pollutants | References |
|----------------|--|--------------|---|-------------------|
| 1. | Guar gum-poly(acrylic acid-aniline) based interpenetrated network hydrogel | 10 | Methylene blue | [64] |
| 2. | Guar gum–polyacrylamide network hydrogel | 7 | Crystal violet and Azure B | [65] |
| 3. | Guar gum based acrylic acid hydrogel | 10 | Methylene blue | [66] |
| 4. | Guar gum/borax hydrogel | Maximum at 9 | Aniline dye | [67] |
| 5. | Guar gum based bentonite clay hydrogel | 2 | Cr(VI) | [68] |
| 6. | Guar gum-polyacrylamide network hydrogel | Maximum at 7 | Cu(II), Ni(II), Pb (II), and Zn(II) | [69] |
| 7. | Polyacrylamide/guar gum graft copolymer hydrogel | 3 | Cr (VI) | [70] |
| 8. | Guar gum-cl-N,N-methylenebis acrylamide hydrogel | 7 | Cu(II) | [71] |
| 9. | Guar gum based silica nanocomposite hydrogel | 9 | Cd(II) | [72] |

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